

PATENT COOPERATION TREATY

PCT
NOTIFICATION OF ELECTION
(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Commissioner
US Department of Commerce
United States Patent and Trademark
Office, PCT
2011 South Clark Place Room
CP2/5C24
Arlington, VA 22202
ETATS-UNIS D'AMERIQUE
in its capacity as elected Office

Date of mailing (day/month/year) 02 February 2001 (02.02.01)	
International application No. PCT/GB00/01186	Applicant's or agent's file reference N.76065A
International filing date (day/month/year) 28 March 2000 (28.03.00)	Priority date (day/month/year) 29 March 1999 (29.03.99)
Applicant HARDY, Bryan, Anthony et al	

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:

30 October 2000 (30.10.00)

☐ in a notice effecting later election filed with the International Bureau on:
2. The election ☒ was
☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

<p>The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland</p> <p>Facsimile No.: (41-22) 740.14.35</p>	<p>Authorized officer Juan Cruz</p> <p>Telephone No.: (41-22) 338.83.38</p>
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PATENT COOPERATION TREATY

PCT

NOTIFICATION OF RECEIPT OF
RECORD COPY

(PCT Rule 24.2(a))

From the INTERNATIONAL BUREAU

To:

J. A. KEMP & Co

CRESSWELL, Thomas, Anthony

J.A. Kemp & Co. REC'D 31 MAY 2000

14 South Square

Gray's Inn

London WC1R 5LS

ROYAUME-UNI

Action by GWN

Date of mailing (day/month/year) 12 May 2000 (12.05.00)	IMPORTANT NOTIFICATION
Applicant's or agent's file reference N.76065A	International application No. PCT/GB00/01186

The applicant is hereby notified that the International Bureau has received the record copy of the international application as detailed below.

Name(s) of the applicant(s) and State(s) for which they are applicants:

CHROMALLOY UNITED KINGDOM LIMITED et al (for all designated States except US)

HARDY, Bryan, Anthony et al (for US)

International filing date : 28 March 2000 (28.03.00)

Priority date(s) claimed : 29 March 1999 (29.03.99)

Date of receipt of the record copy
by the International Bureau : 18 April 2000 (18.04.00)

List of designated Offices :

AP : GH,GM,KE,LS,MW,SD,SL,SZ,TZ,UG,ZW

EA : AM,AZ,BY,KG,KZ,MD,RU,TJ,TM

EP : AT,BE,CH,CY,DE,DK,ES,FI,FR,GB,GR,IE,IT,LU,MC,NL,PT,SE

OA : BF,BJ,CF,CG,CI,CM,GA,GN,GW,ML,MR,NE,SN,TD,TG

National : AE,AG,AL,AM,AT,AU,AZ,BA,BB,BG,BR,BY,CA,CH,CN,CR,CU,CZ,DE,DK,DM,DZ,EE,ES,
FI,GB,GD,GE,GH,GM,HR,HU,ID,IL,IN,IS,JP,KE,KG,KP,KR,KZ,LC,LK,LR,LS,LT,LU,LV,MA,MD,
MG,MK,MN,MW,MX,NO,NZ,PL,PT,RO,RU,SD,SE,SG,SI,SK,SL,TJ,TM,TR,TT,TZ,UA,UG,US,UZ,VN,
YU,ZA,ZW

ATTENTION

The applicant should carefully check the data appearing in this Notification. In case of any discrepancy between these data and the indications in the international application, the applicant should immediately inform the International Bureau.

In addition, the applicant's attention is drawn to the information contained in the Annex, relating to:

- ☒ time limits for entry into the national phase
- ☐ confirmation of precautionary designations
- ☒ requirements regarding priority documents

A copy of this Notification is being sent to the receiving Office and to the International Searching Authority.

The International Bureau of WIPO
34, chemin des Colombettes
1211 Geneva 20, Switzerland

Facsimile No. (41-22) 740.14.35

Authorized officer:

Ingrid Aulich

Telephone No. (41-22) 358.83.38

INFORMATION ON TIME LIMITS FOR ENTERING THE NATIONAL PHASE

The applicant is reminded that the "national phase" must be entered before each of the designated Offices indicated in the Notification of Receipt of Record Copy (Form PCT/IB/301) by paying national fees and furnishing translations, as prescribed by the applicable national laws.

The time limit for performing these procedural acts is **20 MONTHS** from the priority date or, for those designated States which the applicant elects in a demand for international preliminary examination or in a later election, **30 MONTHS** from the priority date, provided that the election is made before the expiration of 19 months from the priority date. Some designated (or elected) Offices have fixed time limits which expire even later than 20 or 30 months from the priority date. In other Offices an extension of time or grace period, in some cases upon payment of an additional fee, is available.

In addition to these procedural acts, the applicant may also have to comply with other special requirements applicable in certain Offices. It is the **applicant's responsibility** to ensure that the necessary steps to enter the national phase are taken in a timely fashion. Most designated Offices do not issue reminders to applicants in connection with the entry into the national phase.

For detailed information about the procedural acts to be performed to enter the national phase before each designated Office, the applicable time limits and possible extensions of time or grace periods, and any other requirements, see the relevant Chapters of Volume II of the PCT Applicant's Guide. Information about the requirements for filing a demand for international preliminary examination is set out in Chapter IX of Volume I of the PCT Applicant's Guide.

GR and ES became bound by PCT Chapter II on 7 September 1996 and 6 September 1997, respectively, and may, therefore, be elected in a demand or a later election filed on or after 7 September 1996 and 6 September 1997, respectively, regardless of the filing date of the international application. (See second paragraph above.)

Note that only an applicant who is a national or resident of a PCT Contracting State which is bound by Chapter II has the right to file a demand for international preliminary examination.

CONFIRMATION OF PRECAUTIONARY DESIGNATIONS

This notification lists only specific designations made under Rule 4.9(a) in the request. It is important to check that these designations are correct. Errors in designations can be corrected where precautionary designations have been made under Rule 4.9(b). The applicant is hereby reminded that any precautionary designations may be confirmed according to Rule 4.9(c) before the expiration of 15 months from the priority date. If it is not confirmed, it will automatically be regarded as withdrawn by the applicant. There will be no reminder and no invitation. Confirmation of a designation consists of the filing of a notice specifying the designated State concerned (with an indication of the kind of protection or treatment desired) and the payment of the designation and confirmation fees. Confirmation must reach the receiving Office within the 15-month time limit.

REQUIREMENTS REGARDING PRIORITY DOCUMENTS

For applicants who have not yet complied with the requirements regarding priority documents, the following is recalled.

Where the priority of an earlier national, regional or international application is claimed, the applicant must submit a copy of the said earlier application, certified by the authority with which it was filed ("the priority document") to the receiving Office (which will transmit it to the International Bureau) or directly to the International Bureau, before the expiration of 16 months from the priority date, provided that any such priority document may still be submitted to the International Bureau before that date of international publication of the international application, in which case that document will be considered to have been received by the International Bureau on the last day of the 16-month time limit (Rule 17.1(a)).

Where the priority document is issued by the receiving Office, the applicant may, instead of submitting the priority document, request the receiving Office to prepare and transmit the priority document to the International Bureau. Such request must be made before the expiration of the 16-month time limit and may be subjected by the receiving Office to the payment of a fee (Rule 17.1(b)).

If the priority document concerned is not submitted to the International Bureau or if the request to the receiving Office to prepare and transmit the priority document has not been made (and the corresponding fee, if any, paid) within the applicable time limit indicated under the preceding paragraphs, any designated State may disregard the priority claim, provided that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity to furnish the priority document within a time limit which is reasonable under the circumstances.

Where several priorities are claimed, the priority date to be considered for the purposes of computing the 16-month time limit is the filing date of the earliest application whose priority is claimed.

PATENT COOPERATION TREATY

PCT

NOTIFICATION CONCERNING
SUBMISSION OR TRANSMITTAL
OF PRIORITY DOCUMENT

(PCT Administrative Instructions, Section 411)

From the INTERNATIONAL BUREAU

To:

CRESSWELL, Thomas, Anthony
J.A. Kemp & Co. **J.A. KEMP & Co**
14 South Square
Gray's Inn
London WC1R 5LX
ROYAUME-UNI

REC'D 31 MAY 2000

Action by... *Gwn*

Date of mailing (day/month/year) 18 May 2000 (18.05.00)	
Applicant's or agent's file reference N.76065A	IMPORTANT NOTIFICATION
International application No. PCT/GB00/01186	International filing date (day/month/year) 28 March 2000 (28.03.00)
International publication date (day/month/year) Not yet published	Priority date (day/month/year) 29 March 1999 (29.03.99)
Applicant CHROMALLOY UNITED KINGDOM LIMITED et al	

- The applicant is hereby notified of the date of receipt (except where the letters "NR" appear in the right-hand column) by the International Bureau of the priority document(s) relating to the earlier application(s) indicated below. Unless otherwise indicated by an asterisk appearing next to a date of receipt, or by the letters "NR", in the right-hand column, the priority document concerned was submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b).
- This updates and replaces any previously issued notification concerning submission or transmittal of priority documents.
- An asterisk(*) appearing next to a date of receipt, in the right-hand column, denotes a priority document submitted or transmitted to the International Bureau but not in compliance with Rule 17.1(a) or (b). In such a case, the attention of the applicant is directed to Rule 17.1(c) which provides that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity, upon entry into the national phase, to furnish the priority document within a time limit which is reasonable under the circumstances.
- The letters "NR" appearing in the right-hand column denote a priority document which was not received by the International Bureau or which the applicant did not request the receiving Office to prepare and transmit to the International Bureau, as provided by Rule 17.1(a) or (b), respectively. In such a case, the attention of the applicant is directed to Rule 17.1(c) which provides that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity, upon entry into the national phase, to furnish the priority document within a time limit which is reasonable under the circumstances.

<u>Priority date</u>	<u>Priority application No.</u>	<u>Country or regional Office or PCT receiving Office</u>	<u>Date of receipt of priority document</u>
29 Marc 1999 (29.03.99)	9907244.9	GB	15 May 2000 (15.05.00)

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No. (41-22) 740.14.35	Authorized officer Taïeb Akremi <i>T</i> Telephone No. (41-22) 338.83.38
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PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference N. 76065A	FOR FURTHER ACTION		see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.
International application No. PCT/GB 00/ 01186	International filing date (day/month/year) 28/03/2000	(Earliest) Priority Date (day/month/year) 29/03/1999	
Applicant CHROMALLOY UNITED KINGDOM LIMITED et al.			

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 2 sheets.
☒ It is also accompanied by a copy of each prior art document cited in this report.

1. Basis of the report

- a. With regard to the language, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.
- ☐ the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).
- b. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international search was carried out on the basis of the sequence listing :
- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

2. ☐ Certain claims were found unsearchable (See Box I).

3. ☐ Unity of Invention is lacking (see Box II).

4. With regard to the title,

- ☒ the text is approved as submitted by the applicant.
- ☐ the text has been established by this Authority to read as follows:

5. With regard to the abstract,

- ☒ the text is approved as submitted by the applicant.
- ☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the drawings to be published with the abstract is Figure No.

- ☐ as suggested by the applicant.
- ☐ because the applicant failed to suggest a figure.
- ☐ because this figure better characterizes the invention.
- ☒ None of the figures.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 00/01186

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C23C10/04

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C23C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

WPI Data, CHEM ABS Data, EPO-Internal, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DE 12 98 830 B (DEUTSCHE EDELSTAHLWERKE) 3 July 1969 (1969-07-03) ---	
A	DATABASE WPI Section Ch, Week 198146 Derwent Publications Ltd., London, GB; Class M13, AN 1981-84937D XP002141086 & SU 804 715 A (S RANCHES MECH ELEC), 15 February 1981 (1981-02-15) abstract ---	
A	US 4 128 522 A (RICHARD C. ELAM) 5 December 1978 (1978-12-05) ---	
A	GB 2 008 621 A (ROLLS-ROYCE) 6 June 1979 (1979-06-06) -----	

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

30 June 2000

Date of mailing of the international search report

07/07/2000

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Elsen, D

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

CT/GB 00/01186

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
DE 1298830	B	NONE	
SU 804715	A	15-02-1981	NONE
US 4128522	A	05-12-1978	DE 2733908 A 02-02-1978 FR 2359965 A 24-02-1978 GB 1535691 A 13-12-1978 US 4181758 A 01-01-1980
GB 2008621	A	06-06-1979	NONE

PATENT COOPERATION TREATY

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

CRESSWELL, Thomas
J.A. KEMP & CO.
14 South Square
Gray's Inn
London WC1R 5LX
GRANDE BRETAGNE

J. A. KEMP & Co

29 DEC 2000

Action by -----

PCT

NOTIFICATION OF TRANSMITTAL OF
THE INTERNATIONAL PRELIMINARY
EXAMINATION REPORT
(PCT Rule 71.1)

Date of mailing
(day/month/year) 27.12.2000

Applicant's or agent's file reference
N.76065A

IMPORTANT NOTIFICATION

International application No.
PCT/GB00/01186

International filing date (day/month/year)
28/03/2000

Priority date (day/month/year)
29/03/1999

Applicant
CHROMALLOY UNITED KINGDOM LIMITED et al.

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/

 European Patent Office
D-80298 Munich
Tel. +49 89 2399 - 0 Tx: 523656 epmu d
Fax: +49 89 2399 - 4465

Authorized officer

Myers, J

Tel. +49 89 2399-8111



PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)



Applicant's or agent's file reference N.76065A	FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/GB00/01186	International filing date (day/month/year) 28/03/2000	Priority date (day/month/year) 29/03/1999
International Patent Classification (IPC) or national classification and IPC C23C10/04		
Applicant CHROMALLOY UNITED KINGDOM LIMITED et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of 6 sheets, including this cover sheet.
 - ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 2 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☒ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand 30/10/2000	Date of completion of this report 27.12.2000
Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer Piber-Goldbacher, U Telephone No. +49 89 2399 7327 

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB00/01186

I. Basis of the report

1. This report has been drawn on the basis of *(substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments (Rules 70.16 and 70.17).)*

Description, pages:

1-9 as originally filed

Claims, No.:

1-26 as originally filed

Drawings, sheets:

1/2,2/2 as received on 05/06/2000 with letter of 05/06/2000

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/GB00/01186

☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

III. Non-establishment of opinion with regard to novelty, inventive step and industrial applicability

1. The questions whether the claimed invention appears to be novel, to involve an inventive step (to be non-obvious), or to be industrially applicable have not been examined in respect of:

☐ the entire international application.

☒ claims Nos. 20-26.

because:

☐ the said international application, or the said claims Nos. relate to the following subject matter which does not require an international preliminary examination (*specify*):

☒ the description, claims or drawings (*indicate particular elements below*) or said claims Nos. 20-26 are so unclear that no meaningful opinion could be formed (*specify*):
see separate sheet

☐ the claims, or said claims Nos. are so inadequately supported by the description that no meaningful opinion could be formed.

☐ no international search report has been established for the said claims Nos. .

2. A meaningful international preliminary examination report cannot be carried out due to the failure of the nucleotide and/or amino acid sequence listing to comply with the standard provided for in Annex C of the Administrative Instructions:

☐ the written form has not been furnished or does not comply with the standard.

☐ the computer readable form has not been furnished or does not comply with the standard.

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)

Yes: Claims 1-19

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/GB00/01186

	No:	Claims	
Inventive step (IS)	Yes:	Claims	1-19
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-19
	No:	Claims	

2. Citations and explanations
see separate sheet

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/GB00/01186

Re Item III

Non-establishment of opinion with regard to novelty, inventive step and industrial applicability

Claims 20-26 refer merely to the contents of the foregoing claims and examples in some vague and not precisely defined way. This throws doubt on the extent of protection and renders these claims so unclear that no meaningful opinion could be formed (cf. Rule 6.2(a) PCT).

Re Item V

Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

Reference is made to the following documents:

D1: DE 12 98 830 B (DEUTSCHE EDELSTAHLWERKE) 3 July 1969 (1969-07-03)

D2: GB-A-2 008 621 (ROLLS-ROYCE) 6 June 1979 (1979-06-06)

Prior Art:

D1 discloses a layer for protecting a portion of a substrate surface against diffusion coating, said layer comprising a silicate and a metall oxide (cf. Example 2) or a metall oxide and a metall (cf. Example 3).

D2 discloses a layer for protecting a portion of a substrate surface against diffusion coating, said layer comprising zirconium silicate (cf. Example 1) or zirconium oxide and nickel (cf. Examples 2 and 3).

The layers according to D1 or D2 are not reusable.

Novelty:

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/GB00/01186

The subject matter of the independent claim 1 is novel, since none of the cited documents reveals a mask for protecting a portion of a substrate surface against diffusion coating which comprises silica, an inert refractory diluent and a metall.

Inventive Step:

The technical problem solved by the subject matter of the application is to provide a reusable coating mask which does not interact with the substrate surface even at high temperatures and which prevents diffusion coating.

The subject-matter of claim 1 is considered to be inventive, because it solves the aforementioned problem in a way that is not suggested by the prior art.

Since the composition of the mask according to claim 1 is considered to be novel and inventive, the independent process claims 12 and 16, disclosing the preparation of said mask and the diffusion coating of a substrate by use of said mask respectively, are novel and inventive as well.

Dependent claims 2-11, 13-15 and 17-19 only add further features to the subject-matter of the independent claims to which they refer and are therefore also considered to be novel and inventive.

05 JUNE 2000

1/2

Fig.1.

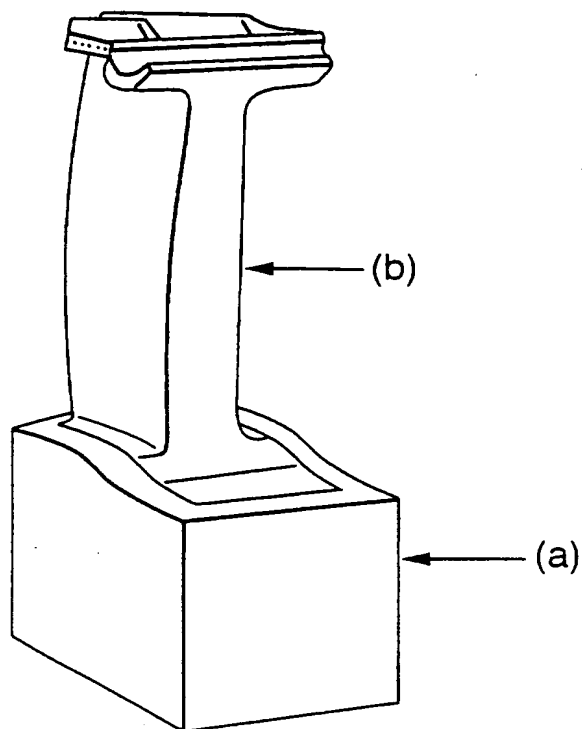
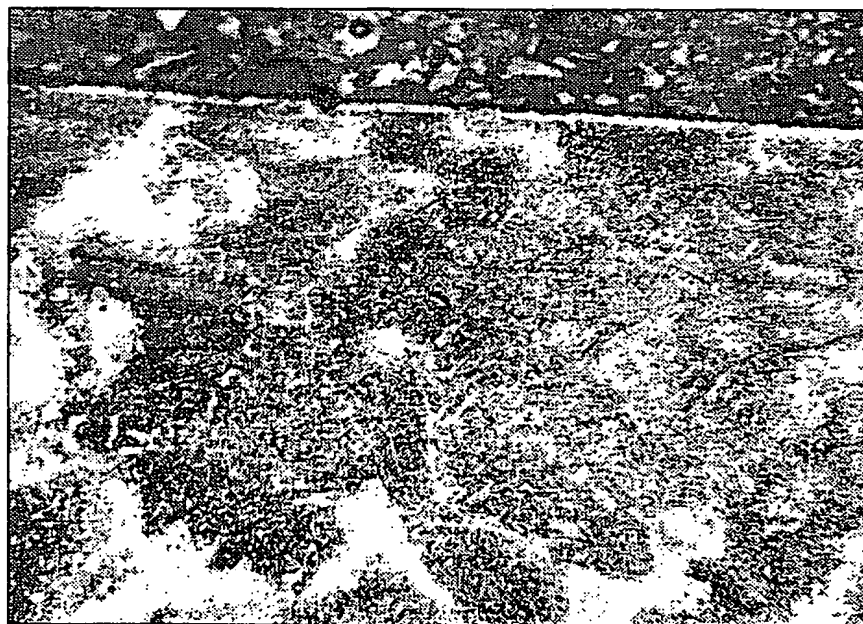
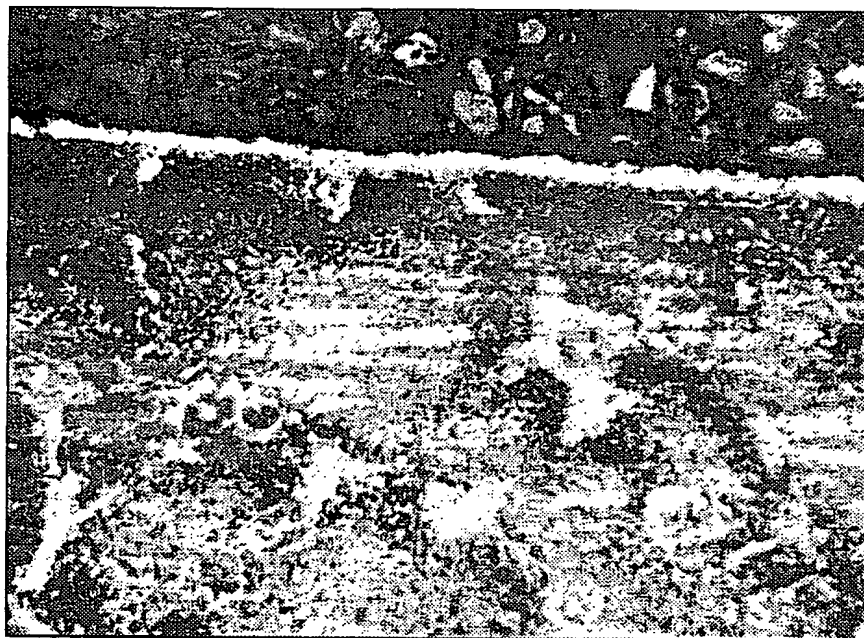


Fig.2.



2/2

Fig.3.



PCTWORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁷ : C23C 10/04	A1	(11) International Publication Number: WO 00/58531 (43) International Publication Date: 5 October 2000 (05.10.00)
(21) International Application Number: PCT/GB00/01186 (22) International Filing Date: 28 March 2000 (28.03.00) (30) Priority Data: 9907244.9 29 March 1999 (29.03.99) GB (71) Applicants (for all designated States except US): CHROMA-LLOY UNITED KINGDOM LIMITED [GB/GB]; Bramble Way, Clover Nook Industrial Estate, Somercotes, Derbyshire DE55 4RH (GB). WADE CERAMICS LIMITED [GB/GB]; Royal Works, Westport Road, Burslem, Stoke-on-Trent, Staffordshire ST6 4AP (GB). (72) Inventors; and (75) Inventors/Applicants (for US only): HARDY, Bryan, Anthony [GB/GB]; 16 Trent Lane, Kings Newton, Derby, Derbyshire DE73 1BT (GB). GODDARD, David, Arthur [GB/GB]; 22 Endon Drive, Biddulph, Stoke-on-Trent, Staffordshire ST6 8NJ (GB). SHAW, Edward, Hugh [GB/GB]; Crow Holt Farm, Basford, Leek, Staffordshire ST13 7DU (GB). (74) Agents: CRESSWELL, Thomas, Anthony et al.; J.A. Kemp & Co., 14 South Square, Gray's Inn, London WC1R 5LX (GB).		(81) Designated States: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>
(54) Title: STOP-OFF FOR DIFFUSION COATING (57) Abstract A mask suitable for protecting a portion of a substrate surface against diffusion coating of the substrate with a metal, which mask comprises a ceramic material comprising silica and an inert refractory diluent and a metal or metal alloy, wherein the metal or metal alloy is one which is reactive with silicon thereby minimising or preventing siliconisation of the substrate with silicon in the ceramic material under conditions of diffusion coating, and which is reactive with the metal being applied by diffusion coating thereby preventing diffusion coating of the portion of the substrate surface it is desired to protect.		

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STOP-OFF FOR DIFFUSION COATING

The present invention relates to a mask for use in diffusion coating, to its preparation and its use in a diffusion coating process. The invention further relates to a composition/mixture of components suitable for use in preparing the mask.

5 Diffusion coating of substrate surfaces, such as high temperature superalloys, to introduce metal into the substrate surface, is typically carried out at high temperatures. Under coating conditions the metal which it is desired to introduce pervades to all substrate surfaces unless special precautions are taken to prevent this. Indeed, in many applications, it is important to restrict coating of the substrate to
10 certain areas. For example, when the substrate is a jet engine turbine blade, it is important that the turbine roots remain uncoated if mounting dimension tolerances are to be maintained.

 A number of methods of masking a substrate surface to prevent diffusion coating have been proposed. Some methods involve the preparation and application
15 of stop-off pastes, slurries or resins. These are typically metal loaded compositions in which the metal serves to react with the metallic coating vapours, thereby preventing metal deposition in unwanted areas. The use of this kind of masking technique is labour and time intensive and requires the careful application of the composition to that area of the substrate to be protected, followed by drying of the
20 composition. Often a number of layers of composition need to be applied before diffusion coating. After coating, the mask must be fractured and removed. In this respect, the use of such stop-off compositions is also uneconomical due to their "one off" usage. It has also been observed that the compositions tend to exhibit reduced effectiveness at higher coating temperatures: at elevated temperatures components of
25 the mask composition can interact with the substrate surface to the detriment of the metallurgy of the component.

 As an alternative, it has also been proposed to use plain (non metal-containing) ceramic caps to shield substrate surfaces. Silica based ceramic materials have been used previously. These have the benefit that they may be re-usable but
30 are only effective at lower temperature range short cycle processes because of the danger of siliconisation of the protected area of the substrate due to silicon in the ceramic.

The present invention seeks to overcome these problems by providing a re-usable diffusion coating mask which provides a higher level of protection, which does not interact with the substrate surface even at higher coating temperatures or relatively longer coating cycles, and which minimises consumables, depositing and removal costs.

It has now been found that incorporating a metal or metal alloy into a silica-based ceramic material prevents the siliconisation problem encountered with the previously used plain ceramic caps. This enables the masks to be used at higher temperatures or over longer coating cycles. The metal or alloy used is also capable of reacting with the metallic coating vapours being applied thereby preventing diffusion coating in areas of a substrate protected by such material. The finding that the metal or alloy is able to prevent both siliconisation and diffusion coating is central to the present invention.

Accordingly, the present invention provides a mask suitable for protecting a portion of a substrate surface against diffusion coating of the substrate by metallic vapours during a pack or vapour coating process. This mask comprises a composite material containing silica and an inert refractory diluent and a metal or metal alloy, wherein the metal or metal alloy is one which is capable of reacting with silicon thereby preventing siliconisation of the substrate with silicon from the composite material under conditions of diffusion coating and which is capable of reacting with the metal being applied by diffusion coating thereby preventing diffusion coating of the portion of the substrate surface it is desired to protect.

The composite material usually contains between 5 and 50% by weight metal or metal alloy based on the total weight of the composite material. In a preferred embodiment, the amount of metal or metal alloy is between 10 and 20% by weight. Single metals or metal alloys may be used, or mixtures of different metals and/or metal alloys. When mixtures are used, the total amount of metal and/or metal alloy generally falls within these limits.

The metal or metal alloy is usually present in the ceramic matrix in the form of particles. The particles may vary in size from fine powders to granules depending upon application. Typically, the particles range between 25 and 150 microns. Particles of 75 microns or finer are typically used.

Examples of metals which may be used in practice of the present invention include nickel, cobalt, chromium, molybdenum and tungsten. Of these, the use of nickel or cobalt is preferred, particularly nickel.

Useful metal alloys which may be used include alloys based on combinations of the following metals: nickel, cobalt, chromium, aluminium, molybdenum, tungsten, vanadium, tantalum, titanium and hafnium. Of these, the use of nickel-chromium alloys is preferred.

The composite material is a ceramic which contains silica and an inert refractory diluent. The latter prevents sintering to the surface being masked. Refractory diluents of alumina, aluminosilicates and feldspar (plus trace elements) are typically employed. The use of alumina is preferred. The silica is usually present in the composite material (i.e. excluding the metal or metal alloy) in an amount of at least 5% by weight. The amount of silica does not usually exceed 30% by weight based on the weight of the composite material. More typically, the amount of silica is from 10 to 15% by weight. The proportion of silica in the composite can be adjusted to optimise the structural integrity of the mask although here it will be appreciated that any variation in silicon content may require variation also in the content of metal or metal alloy required to inhibit siliconisation. Determination of the amount of metal or alloy for a particular silicon content is within the ability of one skilled in the art.

In a preferred embodiment of the invention, the ceramic is an aluminosilicate. Thus, the masks may be conveniently prepared using clays. Useful clays are commercially available and include Puraflow-DM and Bentonite. As a consequence of using a clay, the ceramic will also include other compounds and minerals commonly found in clays. In an embodiment of the invention the mask comprises 10 to 20% by weight nickel dispersed in an aluminosilicate ceramic matrix.

The metal or alloy in the mask must be in reduced form to ensure that it is available for reaction both with the silicon present in the composite material and with the metal which is being applied by diffusion coating. This requirement has particular implications with respect to how the mask is prepared. Thus, the present invention further provides a process for preparing the mask, which process comprises mixing the metal or metal alloy with a ceramic material containing silica and an inert

refractory diluent, shaping the resultant mixture into a desired configuration to form a blank, and then either:

(a) firing the blank in a reducing atmosphere to prevent oxidation of the metal or metal alloy; or

5 (b) firing the blank in an oxidising atmosphere followed by treatment in a reducing atmosphere to reduce the metal or metal alloy.

In one embodiment of this process the blank is fired in a reducing atmosphere, such as hydrogen or other reducing atmosphere. Firing typically takes place at a temperature of between 1150 and 1300°C for a period of time of from 30
10 minutes to 3 hours at temperature.

In the other embodiment of the process, the blank is initially fired in a conventional manner, i.e. without special steps to prevent oxidation of the metal or metal alloy. In this case, the initial firing typically also takes place at a temperature of between 1150 to 1300°C for a period of time of 30 minutes to 3 hours at
15 temperature. Subsequent to this firing, a conditioning treatment is then necessary in order to achieve reduction of the metal or metal alloy. This reduction may be achieved by heat-treatment in a reducing atmosphere (e.g. hydrogen or other) at a temperature of between 900 and 1200 °C for a period of at least one hour.

The conditions required to reduce the metal or metal alloy to the desired
20 extent may be determined easily. For example, this may be done on a trial and error basis by considering the effectiveness of the mask in the diffusion coating process. In this way, it is also possible to optimise the amount of metal or metal alloy which needs to be present in the mask.

In certain cases the extent to which the metal or alloy has been reduced can be
25 assessed visually as the colour of the metal or alloy changes with oxidation/reduction. For instance, when the mask contains nickel reduction leads to a colour change of the mask from green (nickel oxide) to grey (nickel). To achieve effective masking, the metal or alloy should be substantially in reduced form through the entire mask. Thus, for a nickel-containing mask, the grey colour should be
30 observed through any section of the mask.

The present invention also provides a mixture of components suitable for preparing the masks described herein. Thus, the ceramic material and metal or metal

alloy may be provided in ready to use granulate form.

Caps may be formed by conventional techniques such as wet pressing using a suitable die or by other ceramic forming methods. The caps so-formed may then be fired as described above.

5 The masks of the present invention may be used in diffusion coating of aluminium (aluminising) or chromium (chromising), more typically aluminium. The masks may be used in the coating of a variety of components but are expected to have particular usage in the diffusion coating of turbine blades, for example of jet engines, where it is desired to prevent coating of the blade root. Jet engine turbine
10 blades are typically formed from nickel-based superalloys, and when applied to such components, the metal present in the mask is usually nickel or a nickel-based alloy.

Typically, the mask is provided in the form of a cap which is fitted over the part of the substrate to be protected. Such an embodiment is illustrated in Figure 1 which shows a cap (a) fitted to the root of a jet engine turbine blade (b). In this
15 embodiment, the fit of the cap does not have to follow the exact profile of the area being protected although the cavity of the cap into which the substrate (component) fits should be as well-fitting as manufacturing constraints permit. The gap between the substrate and the cap is typically 0.5 mm or less, preferably 0.25 mm or less. If there is insufficient gap, the substrate may become wedged in the cap and thus be
20 difficult to remove without damaging the cap which is, of course, intended to be re-usable. It is important when preparing the cap for a substrate that contraction/expansion of the cap and substrate during coating be taken into account. Shrinkage of the cap during firing should also be accounted for. If the cavity of the cap as prepared is too small, this may be remedied by machining.

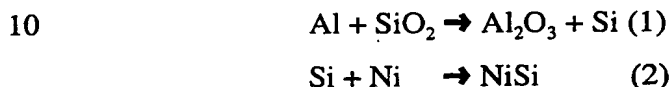
25 The masks of the invention may be used in conventional diffusion coating techniques. For example, aluminising may be carried out by a pack process at a temperature of from 800 to 1050°C for from 1 to 20 hours at temperature, for instance, aluminising at 875°C for 20 hours would be a typical coating cycle.

30 The masks of the invention have the advantage of being re-usable, and may be employed on multiple occasions before their mechanical or protective integrity is diminished to below a useful level.

The basis for the present invention is the choice of a metal or metal alloy

which will react with silicon in the composite and with the metallic coating vapours. With reference to the use of nickel as metal and aluminium as the diffusion coating, the principle underlying the invention is believed to be as follows.

5 The aluminising operation causes dissociation of silicate bonds in the ceramic. The reaction (1) is believed to be oxidation of aluminising vapour to alumina coupled with silica reduction. The silica is then incorporated into the nickel particles forming nickel silicide (NiSi) (2). The latter reaction removes potentially active silicon from the system thereby preventing the siliconisation problem associated previously with plain ceramic masks.



Depletion of silicate bonding within the ceramic tends to reduce the strength of the mask although this is not sufficient to prevent the mask being used on several occasions with effectiveness intact.

15 Some surface depletion in the substrate of elements such as aluminium, chromium and titanium in the area protected by the mask may occur, but this is only to an extent similar to the use of conventional stop-off slurry techniques. This effect may be minimised by including in the ceramic material a metal alloy (e.g. Ni-Cr) at the expense of, or in addition to, pure metal.

20 The invention will now be illustrated by the following non-limiting examples.

Example 1

A ceramic material having the following composition (approx.) was blended with 20% by weight of 99.8% pure nickel powder, at least 40% of which passed through a 38 micron (400 mesh) sieve.

25	Alumina	84%
	Titania	0.02%
	Silica	10.7%
	Ferric oxide	0.26%
	Lime	3.14%
30	Magnesia	1.09%
	Potash	0.24%
	Soda	0.23%

The so-blended material was formed into caps designed to fit the root end of an H.P. turbine blade in MarM002 material. This was done by pressing the mixture using a die of the desired configuration. The caps were then "fired" at a temperature of 1220 °C for 2 hours at temperature. The resultant caps were coloured green due to the presence of nickel in oxidised form. The caps were subsequently treated in a reducing atmosphere (hydrogen) at a temperature of 1100 °C for one hour. The green colour changed to grey indicating reduction to nickel.

The caps were then used to protect the blade roots during pack aluminising for 20 hours at 875 °C. After removal of the caps, the metallurgy of the protected roots was analysed. No evidence of aluminising or siliconising was observed and the level of surface denudation was at least equivalent to that found using conventional stop-off slurries. Figure 2 shows the level of surface denudation on a blade surface protected with the subject invention. Figure 3 shows the level of surface denudation on a blade surface protected using a conventional slurry technique.

Example 2

Adopting the same procedure as Example 1, caps were prepared by blending a ceramic material having the composition (approx.) given below with 10% by weight of 200 mesh 99.8% pure nickel powder, at least 40% of which passed through a 38 micron (400 mesh) sieve.

20	Alumina	85.58%
	Titania	0.13%
	Silica	13.87%
	Ferric oxide	0.29%
	Lime	0.08%
25	Magnesia	0.11%
	Potash	0.36%
	Soda	0.57%

The caps were used to protect the roots of MarM002 turbine blades during aluminising at 875 °C for 20 hours. After the caps were removed and the root structure analysed, identical results to Example 1 were observed.

Example 3

Example 1 was followed to prepare caps with and without nickel addition.

Both types of cap were fired at 1220 °C for 2 hours at temperature followed by reductive conditioning at 1100 °C for 1 hour. The caps were then used as stop-offs on a CMSX4 material (a nickel-cobalt superalloy) during aluminising for 20 hours at 875 °C. After this the metallurgy of the protected surface was analysed. The caps without nickel led to substantial siliconising of the substrate surface. In contrast, no siliconising was observed for the caps containing nickel in accordance with the present invention.

Example 4

A ceramic material including nickel powder (75 micron (200 mesh) to 38 micron (400 mesh)) and having the following composition (approx.) was prepared.

Alumina	71.31%
Titania	0.10%
Silica	11.55%
Ferric oxide	0.24%
Lime	0.06%
Magnesia	0.09%
Potash	0.30%
Soda	0.48%
Nickel	15.87%

This composition was pressed into a cap designed to fit the root end of a MarM002 jet engine turbine blade. The cap was then fired and reduced as in Example 1. On fitting the cap to the root of the blade the gap between the wedge faces of the blade and the cap was found to be 0.25 mm.

The capped-blade was then placed in a pack aluminising retort for 20 hours at 875 °C. After this, the cap was removed and the root of the blade examined. It was clear from visual inspection that the area of the blade protected by the cap had not been aluminised or siliconised. Sections taken through the root for micro-examination confirmed this and that there was a minimum level of denudation. The same cap was re-used on a further four occasions with similarly acceptable results.

Example 5

A similar cap/blade combination to that used in Example 4 was subjected to aluminising at 1100 °C for three hours. Visual appearance again suggested that the

cap had prevented any aluminising of the root, and this was confirmed by micro-examination. There were no signs of siliconisation. There was a slight increase in surface denudation relative to Example 4, but this was to be expected in view of the higher aluminising temperature.

CLAIMS

1. A mask suitable for protecting a portion of a substrate surface against diffusion coating of the substrate by metallic vapours during a pack or vapour coating process which mask comprises a composite material containing silica and an inert refractory diluent and a metal or metal alloy, wherein the metal or metal alloy is one which is capable of reacting with silicon thereby preventing siliconisation of the substrate with silicon from the composite material under conditions of diffusion coating and which is capable of reacting with the metal being applied by diffusion coating thereby preventing diffusion coating of the portion of the substrate surface it is desired to protect.
2. A mask according to claim 1, wherein the metal or metal alloy is present in the composite material in an amount of 5 to 50% by weight based on the total weight of the mask.
3. A mask according to claim 2, wherein the metal or metal alloy is present in the ceramic in an amount of 10 to 20% by weight based on the total weight of the mask.
4. A mask according to any one of the preceding claims, wherein the metal is selected from nickel, cobalt, chromium, molybdenum and tungsten.
5. A mask according to claim 4, wherein the metal is nickel.
6. A mask according to any one of claims 1 to 3, wherein the metal alloy is an alloy based on a combination of metals selected from nickel, cobalt, chromium, aluminium, molybdenum, tungsten, vanadium, tantalum, titanium and hafnium.
7. A mask according to claim 6, wherein the metal alloy is a nickel-chromium alloy.
8. A mask according to any one of the preceding claims, wherein the inert refractory diluent comprises alumina, aluminosilicate or feldspar.
9. A mask according to claim 8, wherein the composite material comprises an aluminosilicate ceramic.
10. A mask according to any one of claims 1 to 5, which comprises from 10 to 20% by weight nickel dispersed in an aluminosilicate ceramic matrix.
11. A mask according to any one of the preceding claims in the form of a diffusion coating cap.

12. A process for preparing a mask as defined in any one of claims 1 to 10, which process comprises mixing the metal or metal alloy with a ceramic material containing silica and an inert refractory diluent material, shaping the resultant mixture into a desired configuration to form a blank, and then either:

5 (a) firing the blank in a reducing atmosphere to prevent oxidation of the metal or metal alloy; or

(b) firing the blank in an oxidising atmosphere followed by treatment in a reducing atmosphere to reduce the metal or metal alloy.

10 13. A process according to claim 12, wherein the blank is in the shape of a cap.

14. A process according to claim 12 or 13, wherein in (a) the blank is fired at a temperature of from 1150 to 1300°C for from 30 minutes to 3 hours at temperature.

15 15. A process according to claim 12 or 13, wherein in (b) the blank is fired in an oxidising atmosphere at a temperature of from 1150 to 1300°C for from 30 minutes to 3 hours at temperature followed by treatment in a reducing atmosphere at a temperature of from 900 to 1200°C for a period of at least 1 hour.

20 16. A process for diffusion coating with a metal a selected portion of a substrate surface, which process comprises masking the substrate surface except for the portion to be coated with a mask as defined in any one of claims 1 to 11, subjecting the substrate to diffusion coating with the metallic vapour, and removing the mask from the substrate surface.

25 17. A process according to claim 16, wherein the metal which is being applied by diffusion coating is aluminium or chromium.

18. A process according to claim 16 or 17, wherein the substrate is a turbine blade and the portion of the blade protected against diffusion coating is the blade root.

19. Use of a mask as defined in any one of claims 1 to 11, to protect the surface of a substrate in a diffusion coating process.

30 20. A mixture suitable for use in preparing a mask as claimed in claim 1, which mixture is as defined in claim 12.

21. A mask according to claim 1 substantially as hereinbefore described

with reference to any of the foregoing Examples.

22. A process according to claim 12, substantially as hereinbefore described with reference to any of the foregoing Examples.

5 23. A mask according to claim 1, when prepared by a process as claimed in any one of claims 12 to 15 or 22.

24. A diffusion coating process according to claim 16, substantially as hereinbefore described with reference to any of the foregoing Examples.

25. Use according to claim 19, substantially as hereinbefore described.

10 26. A mixture according to claim 20, substantially as hereinbefore described.

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Fig.1.

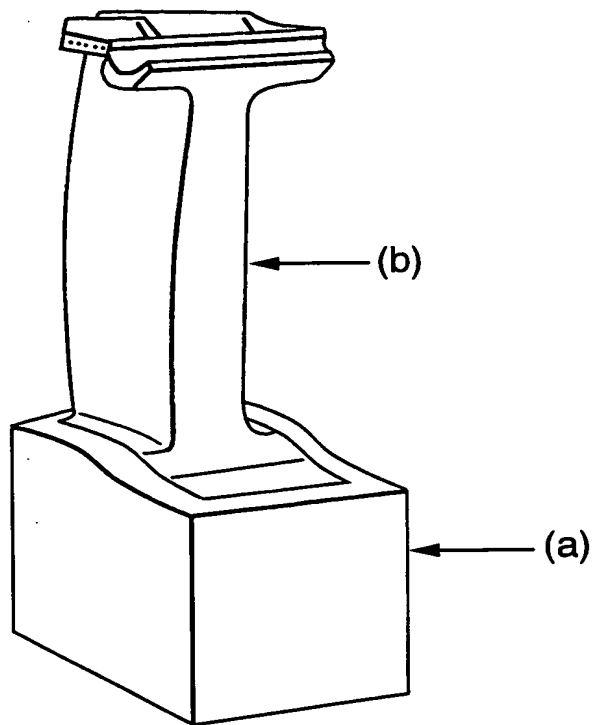
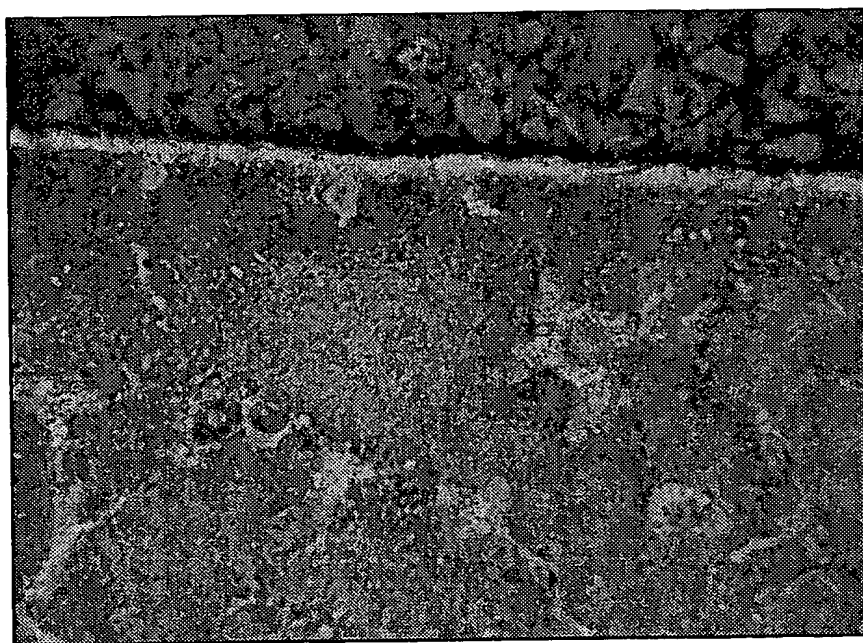


Fig.2.



Fig.3.



INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 00/01186

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C23C10/04

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C23C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

WPI Data, CHEM ABS Data, EPO-Internal, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DE 12 98 830 B (DEUTSCHE EDELSTAHLWERKE) 3 July 1969 (1969-07-03) ---	
A	DATABASE WPI Section Ch, Week 198146 Derwent Publications Ltd., London, GB; Class M13, AN 1981-84937D XP002141086 & SU 804 715 A (S RANCHES MECH ELEC), 15 February 1981 (1981-02-15) abstract ---	
A	US 4 128 522 A (RICHARD C. ELAM) 5 December 1978 (1978-12-05) ---	
A	GB 2 008 621 A (ROLLS-ROYCE) 6 June 1979 (1979-06-06) -----	

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☒ Patent family members are listed in annex.

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

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